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**“पुराने को छोड़ नये के तरफ”**

Jawaharlal Nehru

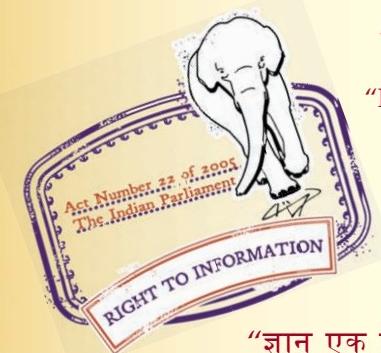
“Step Out From the Old to the New”

IS 10688 (1983): Method for proving gas tightness of vacuum plant [MED 17: Chemical Engineering Plants and Related Equipment]

**“ज्ञान से एक नये भारत का निर्माण”**

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Bhartṛhari—Nītiśatakam

“Knowledge is such a treasure which cannot be stolen”





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*Indian Standard*  
**METHOD FOR**  
**PROVING GAS-TIGHTNESS OF VACUUM PLANTS**

**1. Scope** — This standard describes the following methods for proving gas-tightness of vacuum plants:

- a) Pumping method;
- b) Pressure rise method; and
- c) Mass spectrometer method.

**2. Units** — The unit of leakage rate employed in this standard is litre per second.

**3. Symbols** — The following symbols shall be used in the illustrations :

Valve



Throttle or control valve



Calibrated reference leak



#### 4. Principles of Proving Methods

**4.1 Pumping Method** — The pumping method is an absolute method and the 'leakage throughput' is directly measured. It is applicable to plants in which very low pressures are not required and a moderate leakage rate is tolerable.

**4.2 Pressure Rise Method** — It is an absolute method and the rate of pressure rise is measured directly. This method is applicable to plants with any degree of vacuum-tightness. It is more sensitive than the pumping method, and in practice, less sensitive than search gas method. The disadvantage is that it requires time to provide enough results to determine the true leakage rate. This is especially true if the degassing rate from the walls of the vessels is too high. The advantage is that only commonly used apparatus is required. This method may be used to calibrate a reference leak for the search gas method.

**4.3 Mass Spectrometer Method** — In order to determine the total leakage rate the plant under test is completely surrounded by the search gas. A hood or an envelope may be used for this purpose. Sections of large plants may be hooded, the separate leakage rates being added to obtain the total leakage. If the plant under test is leaking, the air flowing through the leak is replaced by search gas, a partial pressure of search gas builds up. A leakage detector responds preferentially to this gas and indicates its entry into the system. The hood should be made of anti-static material, that is, it should not retain a charge of electricity. Search gases, such as hydrogen are combustible in air and a spark could result in an explosion.

The search gas used for proving gas-tightness must differ from any gas already present in any appreciable amount in the plant or its surroundings and must not cause any chemical reaction, corrosion, contamination within the plant. The precaution against explosion shall be taken, whatever search gas is used. The search gases commonly employed are hydrogen, carbon dioxide, nitrous oxide, organic halides, butane and certain inert gases, in particular helium, depending on the method.

#### 5. Pumping Method

**5.1 Apparatus** — The apparatus consist of the following ( see Fig. 1 ) :

- a) Vacuum gauge, for example, mercury manometer,
- b) Mechanical vacuum pump with pumping capacity in excess of the specified leakage rate,
- c) Surge vessel of suitable capacity to reduce pumping pulsation in the flow to an acceptable level,
- d) Gas measuring devices, for example, Gasometer, gas meter ( w type ) or flowmeter,

- e) Stop-watch or clock (not required with flowmeter),
- f) Barometer (not required with gauges measuring absolute pressure).

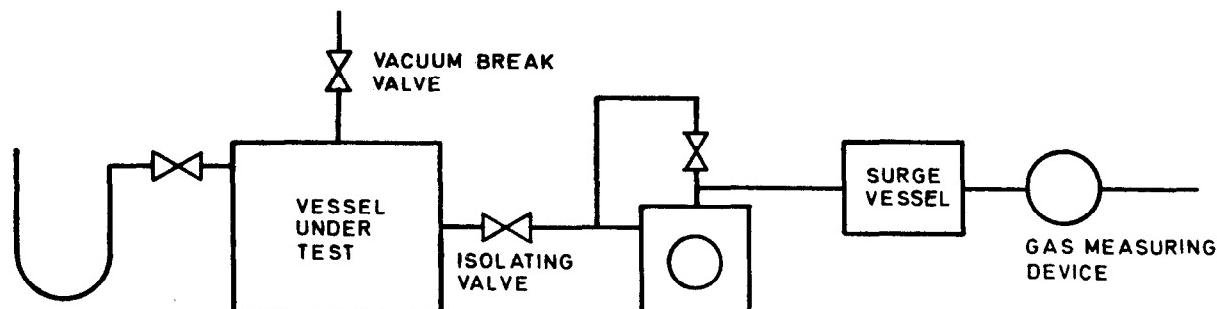


FIG. 1 PUMPING METHOD

**5.2 Procedure** — The pumping system is checked to ensure that there are no significant leaks by closing the isolating and by-pass valves and verifying that there is no indication on the gas measuring device. The pressure in the assembly is reduced; any major leakage at the flanges will become evident due to failure to reach the specified pressure and may be located by suitable means, for example, by external application of solvents like either, carbon tetrachloride. These major leaks are modified before proceeding with the test.

The pressure in the assembly shall be reduced to the specified pressure within the limits  $^{+0}_{-0.07}$  bar if necessary, adjusting the by-pass valve. When steady conditions have been obtained, that is, when successive flow rates taken at suitable intervals do not differ by more than 10 percent, the rate of leakage shall be determined from the mean of the last two readings. Air shall then be allowed to enter the system by opening the vacuum break valve and a second test run shall be carried out by repeating the procedure.

**5.3 Sensitivity** — The sensitivity depends on the range of the flowmeter used. The lower limit of pressure for which the pumping method is useful is 0.035 bar. This smallest detectable leak is of the order of 0.008 bar, litre/second, but it will depend upon the sensitivity of the flow meter.

## 6. Pressure Rise Method

**6.1 Apparatus** — The apparatus consists of the following (see Fig. 2).

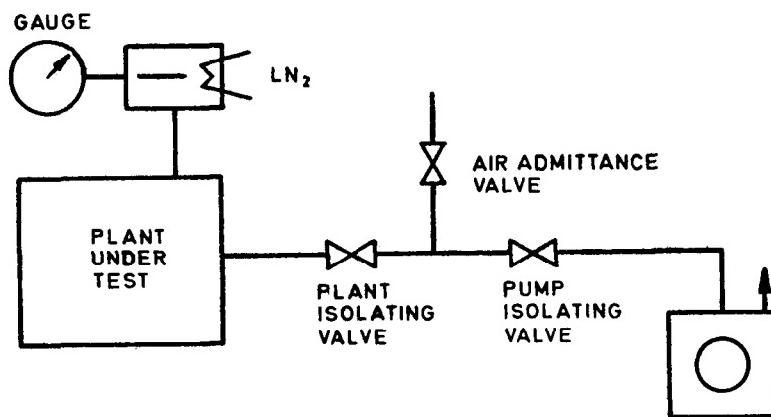


FIG. 2 PRESSURE RISE METHOD

**6.1.1 Vacuum pump** — The performance of the vacuum pump shall be sufficient to exhaust the plant on test to a pressure of the order of working pressure.

**6.1.2 Plant isolating valve** — The closed isolating valve shall have a leak rate small in comparison with the permissible leak rate for the equipment.

### 6.1.3 Vacuum break valve

**6.1.4 Vacuum gauge** — The sensitivity and accuracy of the vacuum gauge shall be appropriate to the rate of pressure rise expected. For measuring small pressure rises (low leakage rates) a gauge of high sensitivity is required but the upper pressure limit of some gauges have a non-linear pressure scale showing a higher sensitivity at lower pressures than at higher pressure; for example, the directly calibrated Mcleod and Pirani type vacuum gauges.

### 6.1.5 Pump isolating valve

**6.1.6 Connecting pipes** — The connecting pipes shall be short and shall not restrict the rate of flow.

### 6.2 Precautions

**6.2.1** For less exacting applications, the leakage rate is either far greater than the outgassing rate or testing may be carried out at pressure where outgassing has virtually ceased (if free water is present, the evaporation will cause a pressure due to water vapour of the order of 0.02 bar at normal temperatures). For more exacting requirements, pumping can be continued until outgassing becomes insignificant in comparison with the leakage. As this condition is reached, the graph of pressure readings against time approximates to a straight line. The attainment of this condition depends on the condition of the plant under test.

**6.2.2** A refinement of technique (other than preliminary heating which can considerably reduce the delay due to outgassing) is the inclusion of a refrigerated trap between the plant under test and the vacuum gauge. With such a trap in use, it is possible to obtain the true leakage rate soon after evacuation. Either the effect of outgassing is eliminated, or even without complete condensation of all vapours, the waiting time is reduced.

**6.2.3** Changes in ambient temperature can be neglected if they occur over a period of the test during which the change in absolute pressure due to leakage is much greater than that due to temperature. Broadly, they should be avoided (or taken into account) from the moment the pressure reaches half of the final reading.

**6.3 Procedure** — When conditions are correct, the isolating valves are closed and the vacuum break valve opened. Assuming that the plant isolating valve is sealed correctly, the pressure will then begin to rise at a rate corresponding to the sum of all the leaks (plus outgassing, which may or may not be neglected).

Simultaneous readings of pressure and time are noted. By taking differences, a series of pressure rises and corresponding time interval can be obtained. As soon as steady conditions are established, the rate of rise per unit time is determined. Steady conditions are considered to exist when successive leak rate is taken as the mean of the last two readings.

The leak rate indicated will include any leakage occurring across the plant isolating valve seating due to atmospheric pressure on its pump side. The validity of the test therefore depends on the tightness of this valve.

**6.4 The Rate of Pressure Rise and Leakage Rate** — The readings shall be in the form of pressure rise in unit time. It is essential to know not only the interval volume of the plant under test but also the volume of the pipe work up to the isolation valve (known as dead volume). Dead volume should show negligible leak rate compared with the permissible leak rate specified for the test. The equivalent pressure rise for the plant only shall be calculated from the observed pressure rise by applying the correction.

$$\text{Equivalent rate of pressure rise} = \frac{\text{Observed rate of pressure rise}}{\text{Plant volume}} \times \frac{(\text{Plant volume} + \text{Dead volume})}{(\text{Plant volume})}$$

This is the information required in the case when leakage is specified as rate of pressure rise in bar per hour. A more common form of expressing the result is a leakage rate in bar litre per second. This is calculated from the observed rate of pressure rise and total volume up to the isolating valve.

$$\text{Leakage rate} = \text{Observed rate of pressure rise} \times (\text{Plant volume} + \text{Dead volume})$$

where

Leakage rate is in bar litre per second, and, volume in litres.

If the dead volume individually tested shows any appreciable pressure rise, its leakage rate may be worked out as :

$$\text{Leakage rate of dead volume} = \text{Observed rate of pressure rise of dead volume} \times \text{Dead volume}$$

The leakage rate of the plant may be estimated by subtracting the leakage rate of the dead volume from the total leakage rate. It is clearly undesirable if the leakage rate of the dead volume becomes comparable to the permissible leak rate of the plant as specified for the test.

**6.5 Sensitivity** — In principle, the pressure rise method may be used to carry out test to any sensitivity apparently depending only on the sensitivity of the pressure gauge used, the volume under test and the time available. In practice the sensitivity is limited by outgassing effects unless auxiliary

equipment is used. For low leakage rates conditions may arise where the outgassing pressure rise is of the same order or even considerably larger than the pressure rise due to leakage and then the results of the tests may not be conclusive.

## 7. Mass Spectrometer Method

### 7.1 Apparatus

**7.1.1** A mass spectrometer leak detector provided with an isolating inlet valve V, which can also be used as a throttle valve to limit the pressure within the instrument.

**7.1.2** A refrigerated trap to protect the mass spectrometer against contaminating vapours.

**7.1.3** A vacuum pumping system auxiliary to that incorporated in the mass spectrometer, with isolating valve. For testing small components an oil-sealed rotary pump may be used as the auxiliary pump. For testing large volumes the pumping down time may be decreased by using vapour pumps. Response and recovery is better with the mass spectrometer connected to the backing space.

**7.2 Pumping Systems for Search Gas Methods** — Search gas methods are conducted under conditions of continuous evacuation. A pumping system is part of the necessary apparatus. It can take any one of the three forms shown in Fig. 3 (that is, system A1, A2, or A3).

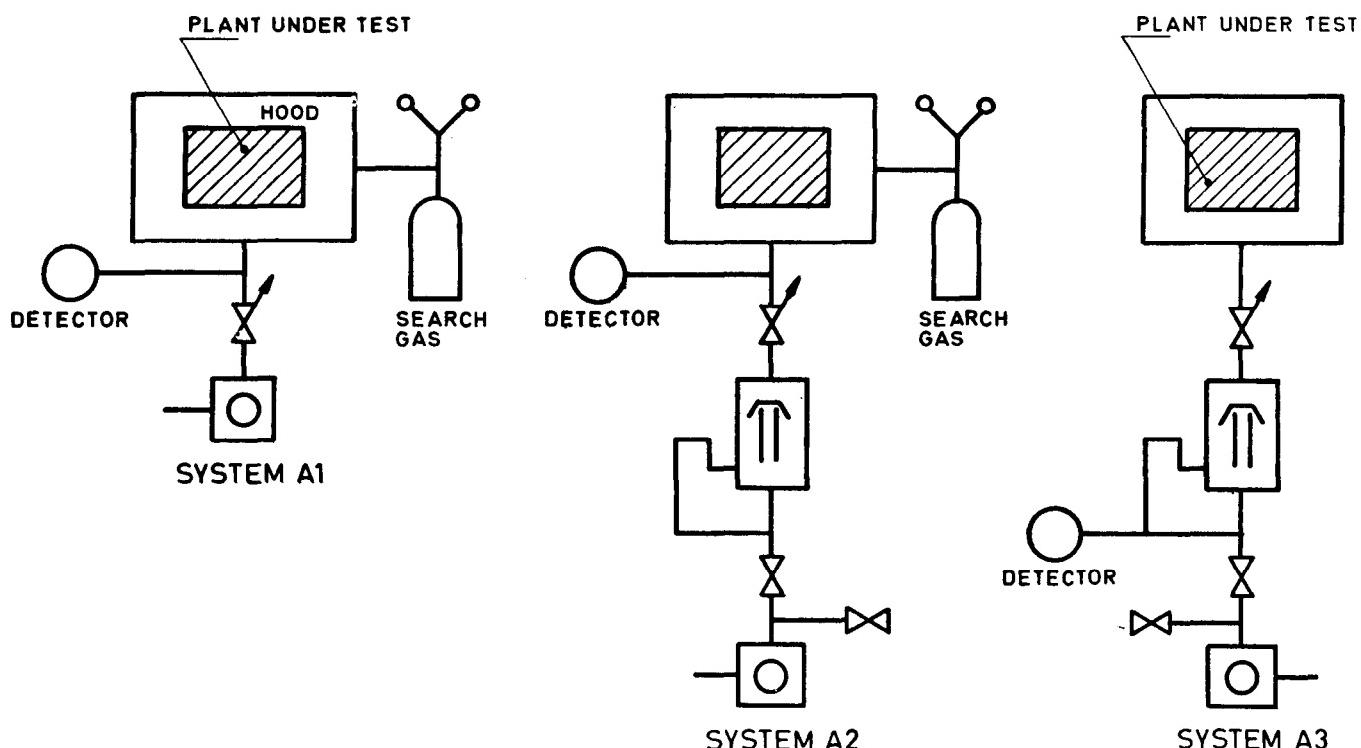


FIG. 3 PUMPING SYSTEM FOR SEARCH GAS METHOD

**7.2.1** System A1 is the simplest. The plant to be tested is pumped only by a rotary pump. The detector is mounted in the plant or connected by a T-joint to the pump. Pressure fluctuations from rotary pump affect this. These are reduced by a control valve, but the valve reduces the pumping speed. The pump, therefore, must have a higher speed. The adjustment of the valve is determined by the gas throughput required at the detector and the pressure needed therein. Sensitivity and speed of response depends on the volume of the plant and the controlled pumping speed.

**7.2.2** In system A2, the plant to be tested is pumped by a vapour diffusion pump and backed by a rotary pump. The detector again is mounted on the hood or connected by a T-joint to the pump. The pressure fluctuations are reduced here and the working pressure of the detector is lower. This arrangement is particularly used for detectors having low operating pressures. If a number of components have to be tested in succession, it is recommended to use a by-pass roughing line to rough the components to the required backing pressure.

**7.2.3** In system A3, the detector is mounted in the backing space of the vapour pump. This method is useful where the volume of the plant to be tested is large, and the test sensitivity obtainable with detector connected to this volume is small. The detector sensitivity is decreased due to the limited control of pumping speed. Given a suitable pressure condition in the vessel, a leak causing a pressure rise in the vessel causes a much higher pressure rise on the backing side. The detector here is subjected to pressure fluctuation.

A vacuum gauge on the plant under test to indicate when the pressure has been reduced to a value low enough not to injure the hot filament.

*A hood to surround the plant under test* — The material must be anti-static, to avoid explosion danger with hydrogen.

*A supply of search gas* — Helium is used in most mass spectrometers. It is an inert gas, not normally present in the plant under test. Hydrogen may be used but precautions are required to prevent interference from hydrogen formed in the breakdown of hydrocarbons in the ion source. The other heavier gases, for example, argon, require the use of more complex mass spectrometers to resolve them satisfactorily from gases of neighbouring mass numbers.

A calibrated reference leak is connected in one of the ways shown in Fig. 4.

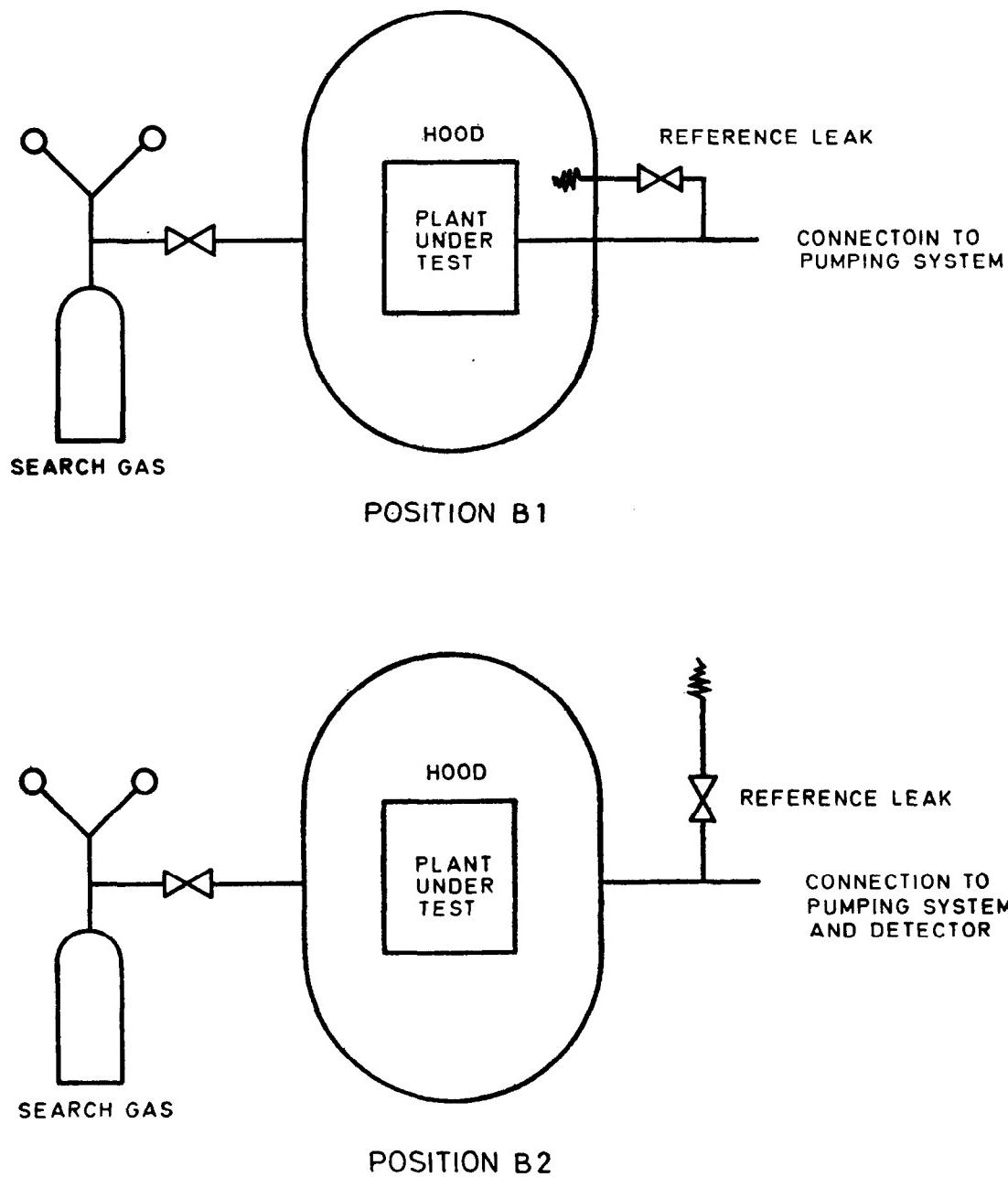


FIG. 4 REFERENCE LEAK POSITIONS

**7.3 Reference Leaks** — Figure 4 shows two positions for a reference leak. In position B1, search gas is applied to the hood, with valve open, and reading A is taken representing leakage through plant and reference leak. Search gas is then applied to the hood with the valve closed and reading B is taken, representing leakage through hood alone. Reading A is repeated to check calibration if the response is linear. The Leakage through plant is  $\frac{B}{A-B} \times$  reference leak.

In position *B2*, search gas is first applied to it and reading *C* taken in detector. Then the search gas is applied to the hood and reading *D* is taken. Reading *C* is repeated to check calibration. The leakage through the plant is  $D/C \times$  reference leak.

Position *B1* gives better results of the concentration of the search gas in all phases of the experiment, but is useful only with a detector with linear response (mass spectrometer and some ionization gauge). Position *B2* with a non-linear detector can show whether the leakage rate of the plant is higher or lower than the reference leak.

Position *B1* is easy to incorporate in a system. In all cases it is necessary to continue the application of the search gas until the reading settles down to a steady value.

**7.4 Precautions** — The hood material shall be anti-static. The refrigerated trap is required to remove condensable vapour contaminants from the inert gas. The amount of contaminants present, depends on the cleanliness of the plant being tested. Any organic vapours entering the ion source would tend to foul the source, mainly by decomposition, and prevent operation of the leak detector with maximum sensitivity. Also, hydrogen from vapour decomposition could lead to a spurious response. It is, therefore, preferable if components to be tested are clean. The refrigerant level in the trap shall be maintained.

The hood and the reference leak must be adequately purged to ensure that the search gas concentration is as intended. Preliminary evacuation of the hood may be helpful.

Equilibrium shall be restored between readings.

#### 7.5 Procedure

- The plant is evacuated by the auxiliary pumping system (Fig. 5 and 6).
- When the pressure is low enough to be safe for the mass spectrometer the gas inlet valve  $V_1$  is slowly opened and valve  $V_2$  is slowly closed, both to the greatest extent consistent with the maintenance of low enough pressure. The degree of opening of valves  $V_1$  and  $V_2$  depends on the pressure condition at the mass spectrometer inlet. For highest sensitivity  $V_2$  will be fully closed and  $V_1$  fully opened. The larger the evacuated volume the less this is likely to be possible.

**7.6 The Leak Rate** — The leak rate is obtained from a calibration of the detector by comparison with the reference leak. The linear characteristics of the instrument make it possible to determine a leakage rate that differs from that of the reference leak.

**7.7 Working Principle** — The special mass spectrometers which have been developed for vacuum testing are essentially self contained instruments incorporating a pumping system, an ion source, an analysing system and an ion collector, with associated electrical supplies and amplifiers.

The process involves the separation of search gas ions from ions of all other gases which may be present in the evacuated volume (air, water vapour). The search gas which has passed

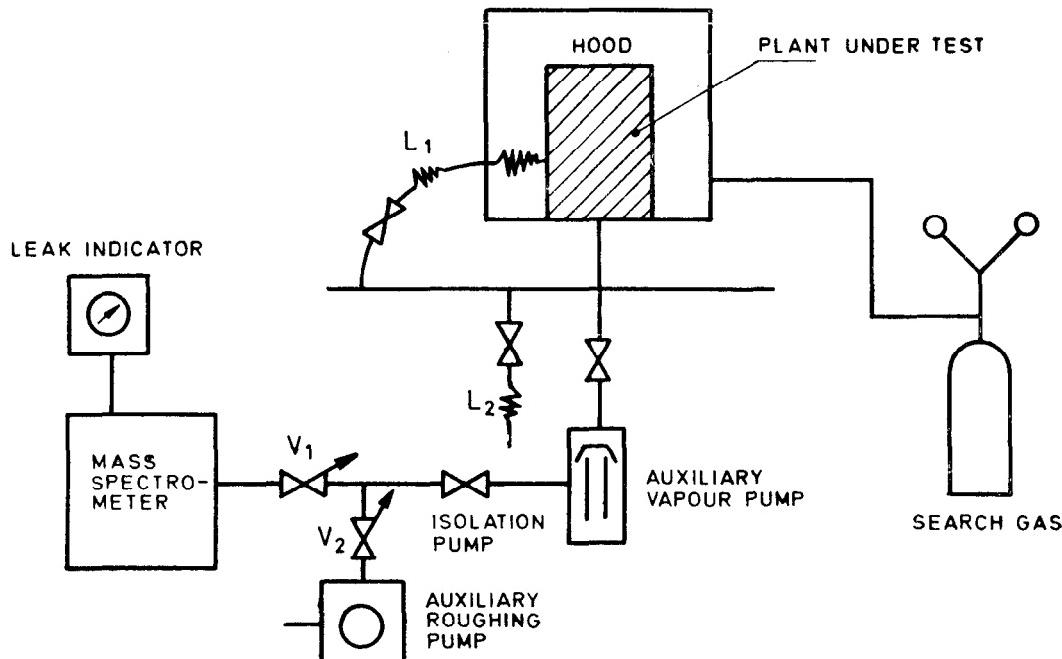


FIG. 5 SYSTEM FOR LARGE VOLUME

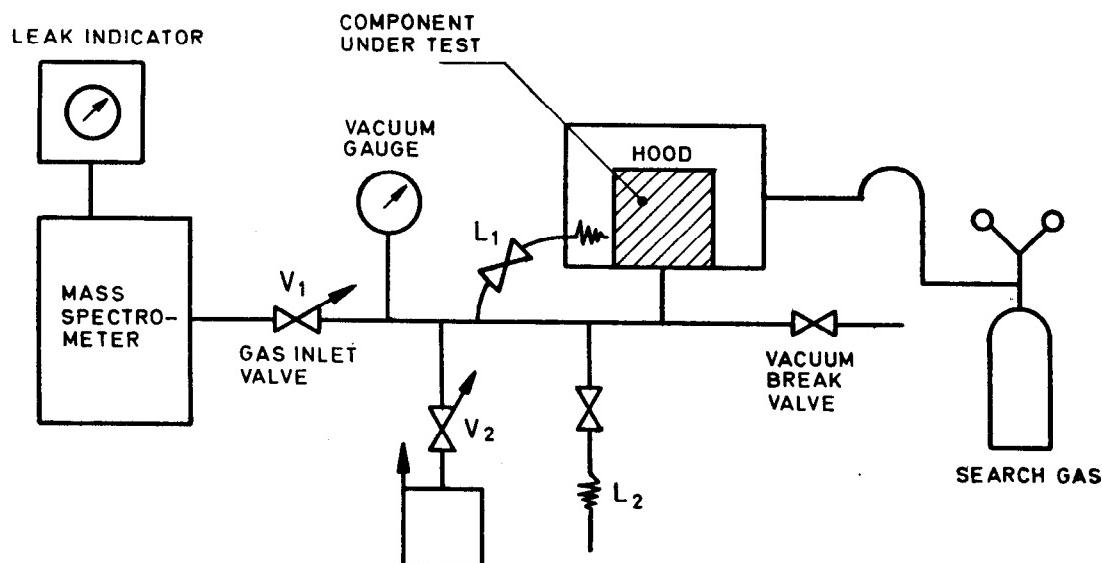


FIG. 6 SYSTEM FOR SMALL VOLUME

through the leak enters the mass spectrometer ion source where it is ionized by an electron beam. The analysing system may be based on a number of different principles, some of which are best suited for differentiating between ions of low mass numbers. Mass spectrometer leak detectors are in fact based on helium as the search gas though hydrogen and argon have also been used in some cases. The advantage of an instrument designed for detecting a gas with a low mass number is that the construction of the mass analysed and associated electrical supplies may be much simpler than would be required in any analytical mass spectrometer covering a range of higher mass numbers. After passing through the analysing section the ions of the search gas impinge on the ion collector plate while ions of other gases that may be present are deflected so that they do not impinge on the collector plate, creating an electric current which is amplified to give an indication on the leak indicator.

The pumping system incorporated in the instrument consists of small vapour pump and mechanical backing pump designed primarily for the continuous evacuation of the spectrometer and not intended for evacuating the articles to be tested. The gas inlet tube on the ion source is usually connected via a refrigerated trap to an inlet valve which serves to connect the mass spectrometer to the plant under test.

The ion source contains a thermionic cathode, usually in the form of very hot filament, the life of which is reduced by continuous operation at higher pressure ( $0.007 - 0.014$  m bar). It may fail altogether if operated instantaneously at a much higher pressure. A protective circuit is therefore often incorporated to switch off the filament whenever the pressure in the spectrometer exceeds a safe operating value.

## 7.8 Sensitivity

### 7.8.1 Factors which affect the sensitivity and response are:

- the physical properties of the search gas which govern the rate at which gas enters the leak,
- the speed at which gas is removed by the pump ( or combination of pumps ),
- the sensitivity of the detector to the particular gas used,
- the volume under test, and
- the position of the sensing element of the detector in the pumping system.

The working pressure affects the locations of the sensing element of the detector, the size and choice of pump and any rough test procedures which may be required.

## EXPLANATORY NOTE

Gas-tightness may be expressed either as the time taken for a given mass of gas to leak through the walls of the equipment or as the time taken for a given pressure change in the equipment. It is usually easier to assess gas-tightness in terms of leakage rate or rate of pressure change, whichever is of more immediate interest.

Leakage rate is general concept, whereas rate of pressure change is specific to the plant volume. Leakage rate has therefore the advantage that it is additive when a number of components are interconnected. The total mass rate of leakage flow into the assembly can be obtained by the summation of the product for each component. This is particularly important for assemblies where the total mass leakage rate is specified or must be known in relation to the controlled flow of a process gas with which the assembly has to operate. An assembly test is of course necessary in all cases to check tightness of joints.

In the preparation of this standard assistance has been derived from BS 3636-1963 'Methods for proving gas tightness of vacuum plants' issued by British Standards Institution.